
**Vacuum technology — Vacuum gauges
— Characterization of quadrupole
mass spectrometers for partial
pressure measurement**

*Technique du vide — Manomètres à vide — Description des
spectromètres de masse quadripolaires pour mesurage de la pression
partielle*

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Contents

	Page
Foreword	v
Introduction	vi
1 Scope	1
2 Normative references	2
3 Terms and definitions	2
4 Symbols and abbreviated terms	3
5 Parameters for which characterization is required or recommended for the different applications	4
5.1 General	4
5.2 General characterization of the QMS	5
5.3 Leak rate measurement and leak rate monitoring (helium leak)	5
5.4 Leak rate monitoring (air leak)	5
5.5 Leak rate monitoring (water leak)	6
5.6 Residual gas analysis	6
5.7 Outgassing rate measurement	7
6 Vacuum systems to characterize QMS	7
6.1 General	7
6.2 Vacuum system for characterization with single gas	8
6.2.1 Continuous expansion system (orifice flow system)	8
6.2.2 Calibration system according to ISO 3567:2012	8
6.2.3 <i>In situ</i> calibration system	8
6.3 Vacuum system for characterization with gas mixtures	9
6.3.1 Continuous expansion system (orifice flow system)	9
6.3.2 <i>In situ</i> calibration system for gas mixture	10
7 Characterization and calibration procedures	10
7.1 General	10
7.2 Mass resolution	11
7.3 Minimum detectable partial pressure (p_{MDPP})	12
7.4 Minimum detectable concentration (C_{MDC})	13
7.5 Dynamic range	13
7.6 Sensitivity and interference effect ratio	14
7.7 Linear response range	15
7.8 Relative sensitivity factor	15
7.9 Fragmentation pattern (cracking pattern)	16
7.10 Outgassing rate of QMS	16
7.11 Pumping speed of QMS	17
8 Measurement uncertainties	17
8.1 General	17
8.2 Uncertainty of mass resolution	18
8.3 Uncertainty of p_{MDPP}	18
8.4 Uncertainty of minimum detectable concentration (C_{MDC})	18
8.5 Uncertainty of dynamic range	18
8.6 Uncertainty of sensitivity	18
8.7 Uncertainty of linear response range	18
8.8 Uncertainty of relative sensitivity factor	18
8.9 Uncertainty of fragmentation factor	18
8.10 Uncertainties of outgassing rate and pumping speed	19
8.11 Long-term stability of characteristic parameters of QMS	19
9 Reporting results	19

Annex A (informative) Estimate of gas composition in the measurement chamber from known gas composition in the reservoir in front of a leak element under different flow conditions	21
Bibliography	23

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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Introduction

Quadrupole mass spectrometers (QMSs) are nowadays used not only in vacuum technology for leak detection and residual gas analysis but also in the process industry as an instrument to provide quantitative analysis in processes and to control processes such as physical and chemical vapour deposition, and etch processes. They are also used for quantitative outgassing rate measurements which are important to characterize vacuum components for critical applications like in the EUV lithography, semiconductor industry or medical instruments.

Total pressure, composition of the gas mixture, settings and the operational history of QMSs, to name a few, have a significant influence on the measured signal, its uncertainty and interpretation. For this reason, it is not possible to calibrate QMS for all its possible applications. Instead, it has either to be calibrated for the special conditions at use or for a standardized condition. It is the purpose of this document to establish such conditions.

There is also a need for standardization in order to enable the users of QMSs to compare the devices of different manufactures and to use the QMS properly.

This document provides standardized calibration procedures for QMSs for some important applications. These have been selected from the results of a survey of the international project EMRP (European Metrological Research Programme) IND12 which was conducted in 2013. This survey included manufacturers, distributors and users of quadrupole mass spectrometers.

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Vacuum technology — Vacuum gauges — Characterization of quadrupole mass spectrometers for partial pressure measurement

1 Scope

This document describes procedures to characterize quadrupole mass spectrometers (QMSs) with an ion source of electron impact ionization and which are designed for the measurement of atomic mass-to-charge ratios $m/z < 300$.

This document is not applicable to QMSs with other ion sources, such as chemical ionization, photo-ionization or field ionization sources and for the measurements of higher m/z , which are mainly used to specify organic materials.

It is well known from published investigations on the metrological characteristics of quadrupole mass spectrometers that their indications of partial pressures depend significantly on the settings of the instrument, the total pressure, and the composition of the gas mixture. For this reason, it is not possible to calibrate a quadrupole mass spectrometer for all possible kinds of use. The characterization procedures described in this document cover the applications of continuous leak monitoring of a vacuum system, leak rate measurement with tracer gas, residual gas analysis and outgassing rate measurements. The user can select that characterization procedure that best suits his or her needs. These characterization procedures can also be useful for other applications.

It is also well known that the stability of several parameters of quadrupole mass spectrometers, in particular sensitivity, are rather poor. Therefore, when a parameter has been calibrated, it needs frequent recalibration when accuracy is required. For practical reasons this can only be accomplished by *in situ* calibrations. To this end, this document not only describes how a quadrupole mass spectrometer can be calibrated by a calibration laboratory or a National Metrological Institute with direct traceability to the System International (SI), but also how calibrated parameters can be frequently checked and maintained *in situ*.

By their physical principle, quadrupole mass spectrometers need high vacuum within the instrument. By reducing dimensions or by special ion sources combined with differential pumping the operational range can be extended to higher pressures, up to atmospheric pressure. This document, however, does not include quadrupole mass spectrometers with differential pumping technology. Therefore, it does not cover pressures exceeding 1 Pa on the inlet flange of the quadrupole mass spectrometer.

This document does not describe how the initial adjustment of a quadrupole mass spectrometer by the manufacturer or by a service given order by the manufacturer should be made. The purpose of such an initial adjustment is mainly to provide a correct m/z scale, constant mass resolution or constant transmission, and is very specific to the instrument. Instead, it is assumed for this document that a manufacturer's readjustment procedure exists which can be carried on-site by a user. This procedure is intended to ensure that the quadrupole mass spectrometer is in a well-defined condition for the characterization.

It is the intention of this document that the user gets the best possible metrological quality from his quadrupole mass spectrometer. From investigations it is known that in most cases this can be achieved in the so called "scan mode". The bar graph may also be of an adequate quality depending on the software used for evaluation of the data taken by the quadrupole mass spectrometer. The trend mode, however, often involves the additional uncertainty that a shift of the peak value position on the mass scale causes a shift in ion current. For this reason, the scan mode is preferable for most of the measurement procedures of this document.

It is not the intent of this document that all the parameters described be determined for each quadrupole mass spectrometer. However, it is intended that the value of a parameter addressed in this document be determined according to the procedure described in this document if it is given or measured (e.g. for an inspection test).

It is assumed for this document that the applicant is familiar with both the operation of quadrupole mass spectrometers and high and ultra-high vacuum technology.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3567:2011, *Vacuum technology — Vacuum gauges — Calibration by direct comparison with a reference gauge*

ISO 14291, *Vacuum technology — Vacuum gauges — Definitions and specifications for quadrupole mass spectrometer*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 14291 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at <https://www.iso.org/bpp>

— IEC Electropedia: available at <http://www.electropedia.org/>

3.1 <https://standards.iteh.ai/catalog/standards/sist/0305c26d-b7cf-48d7-8b8d-105c0b9c6b3b/iso-ts-20175-2018>

matrix gas

gas or gas mixture that makes the major contribution to the total pressure

3.2

equivalent nitrogen pressure

pressure of nitrogen which would produce the same gauge reading as the pressure of gas acting on a vacuum gauge

[SOURCE: ISO 3529-3:2014, 2.3.5, modified.]

Note 1 to entry: Nitrogen equivalent depends on the type of gauge, since the relative sensitivity factor is different for different types. For this reason, the term should be used with the type of vacuum gauge.

3.3

transmission probability

ratio of ion current of a certain mass-to-charge ratio exiting a quadrupole filter of a QMS to the current of ions of the same mass-to-charge ratio entering it

3.4

scan speed

speed as u ($\Delta m/z=1$) per time with a defined number of signal points per u ($\Delta m/z=1$)

3.5

linear response range

partial pressure range over which the non-linearity is within a specified limit

Note 1 to entry: For the purpose of this document the limit is $\pm 10\%$ from the mean value.

Note 2 to entry: The linear response range can also depend on the conversion of the output current signal to a digital value. Sometimes a single digital bit does not quantise the same amount of current at the lower and upper end of the range.

[SOURCE: ISO 14291:2012, 2.2.18, modified – Notes to entry have been added.]

3.6

leak rate measurement

quantitative measurement of a tracer gas through a leak

3.7

leak rate monitoring

continuous monitoring of one or several selected gas species with respect to the normal background in a vacuum system in order to detect a change caused by a leak

EXAMPLE 1 In an accelerator tube, argon is monitored to detect a leak from air.

EXAMPLE 2 In a fusion reactor, water peaks are monitored to detect a leak from the cooling system.

3.8

fragmentation pattern

pattern (i.e. kinds and relative amounts) of ions produced by a given pure gas in a given mass spectrometer under given conditions

Note 1 to entry: This definition does include the isotopic and isomeric distribution of the species.

[SOURCE: ISO 14291:2012, modified – Notes to entry replaced.]

3.9

interference effect ratio

ratio S_i' / S_i where

S_i' is the sensitivity of a specified gas species i of partial pressure p_i present in an interference gas or interference gas mixture;

S_i is the sensitivity at the same value of p_i when only species i is present.

3.10

interference gas

gas species added to a pure gas that may cause an interference effect

3.11

interference gas mixture

mixture of several gas species added to a pure gas that may cause an interference effect

3.12

dynamic range

ratio of the largest signal to the smallest signal within a spectrum

Note 1 to entry: The difference between minimum detectable concentration (C_{MDC}) as defined in ISO 14291 and dynamic range is that for the C_{MDC} it is acceptable to optimize the signal to noise ratio for the minor constituent, while this is not possible for the dynamic range.

4 Symbols and abbreviated terms

Symbol	Designation	Unit
C_{eff}	effective conductance of a duct, effective pumping speed	m ³ /s or L/s
R_{dyn}	dynamic range	1
f	fragmentation factor	1

Symbol	Designation	Unit
I	ion current at partial pressure p	A
I_0	ion current at residual pressure p_0	A
m	molecular mass in atomic mass units	u
M	molecular mass	kg
C_{MDC}	minimum detectable concentration	1
p_{MDPP}	minimum detectable partial pressure	Pa
p	pressure or partial pressure	Pa
p_0	residual pressure or residual partial pressure	Pa
r_x	relative sensitivity for a specified gas species "x" divided by sensitivity S_{N_2} for nitrogen	1
$q_{v,i}$	volume flow rate of species i into a vacuum pump (pumping speed)	m ³ /s or L/s
R	universal gas constant	J mol ⁻¹ K ⁻¹
S	sensitivity (coefficient)	A/Pa
SI	System International	
S_{N_2}	sensitivity for nitrogen	A/Pa
T	temperature	K
T_p	transmission probability	1
z	the ionization state of a molecule	1
Δm	mass resolution as defined in ISO 14291	u
CEM	continuous dynode electron multiplier	
MCP	micro-channel plate	
QMS	quadrupole mass spectrometer	
SEM	secondary electron multiplier	

NOTE The symbol m characterizes the mass of a molecule in u, while m/z characterizes at which position the molecule with mass m appears on the mass scale indicated by the QMS. This is proportional to the mass-to-charge ratio and therefore also to m/z .

5 Parameters for which characterization is required or recommended for the different applications

5.1 General

ISO 14291 requires a certain number of parameters to be stated by a manufacturer for general characterization. This is covered in 5.1. It is also recommended that the general characterization is applied as characterization of an individual QMS and its performance monitored over its lifetime. It is recommended that the parameters described in the following sections are determined to improve accuracy and reliability of the QMS for the specific application mentioned in the section title.

The extent of such characterizations has to be adapted to the application and can usually not be accomplished by a manufacturer for economical reasons.

5.2 General characterization of the QMS

It is required by ISO 14291 that the following parameters are given by the manufacturer for a general characterization:

- a) linear response range for pure nitrogen;
- b) sensitivity for pure nitrogen in the linear response range as a result of measurement a);
- c) minimum detectable partial pressure for helium and nitrogen;
- d) dynamic range.

It is recommended that, in addition, the following parameters are given as part of the specification of the QMS:

- e) minimum detectable concentration for helium in nitrogen (nitrogen partial pressure at around 10 % of the maximum operational pressure or around 10^{-3} Pa, whatever is lower);
- f) mass resolution at $m/z = 4$ and optionally also $m/z = 28$ and 136(Xenon).

NOTE The upper limit of linear response range of conventional QMSs is typically below 10^{-4} Pa except for QMSs designed for pressures higher than 10^{-2} Pa.

5.3 Leak rate measurement and leak rate monitoring (helium leak)

- a) linear response range for pure helium;
- b) sensitivity for pure helium in the linear response range as a result of measurement a);
- c) interference effect of helium within the linear response range for pure helium by introducing nitrogen of partial pressure of 10^{-3} Pa or the typical operational pressure in the application;
- d) linear response range for helium in nitrogen as a result of measurement c);
- e) minimum detectable partial pressure for helium;
- f) minimum detectable concentration for helium in nitrogen (nitrogen partial pressure around 10^{-3} Pa);
- g) dynamic range.

5.4 Leak rate monitoring (air leak)

This type of characterization depends on the specific need of application. In particular, it is important whether a clean UHV system as a high energy accelerator or a system at high vacuum with many constituents (e.g. fusion or plasma reactor) is monitored.

For a clean UHV system it is recommended that the following is measured:

- a) linear response range for nitrogen, oxygen, and argon, each as pure gas;
- b) sensitivity for nitrogen, oxygen and argon in linear response range as a result of measurement a);
- c) fragmentation pattern for nitrogen and oxygen as a result of measurement a);
- d) relative sensitivity factors for oxygen and argon as a result of measurement a);
- e) dynamic range.

For other systems with background in the high vacuum range with a major gas constituent m (e.g. argon), it is recommended that the following is measured:

- 1) sensitivity for the gas to be monitored as air constituent (nitrogen, oxygen, or argon, whichever applies), in the major gas constituent m at its maximum operational pressure (equivalent nitrogen pressure) between partial pressure of 10^{-7} Pa and maximum operational pressure (equivalent nitrogen pressure);
- 2) relative sensitivity factors for the monitoring gas as a result of measurement a);
- 3) fragmentation pattern for the monitoring gas as a result of measurement a);
- 4) minimum detectable concentration for the monitoring gas in the major gas constituent;
- 5) dynamic range.

5.5 Leak rate monitoring (water leak)

- a) Sensitivity for pure water vapour near 10^{-5} Pa;
- b) fragmentation pattern for water vapour as a result of measurement a);
- c) interference effect of water vapour at 10^{-5} Pa by introducing nitrogen or the major constituent of the residual gas in the application at 10^{-3} Pa pressure or the operational pressure in the application;
- d) linear response range for water vapour in nitrogen or the major constituent of the residual gas as a result of measurement c);
- e) fragmentation pattern for water vapour as a result of measurement c);
- f) minimum detectable concentration for water vapour in nitrogen or the major constituent of the residual gas (partial pressure preferably near 10^{-3} Pa or the operational pressure in the application).

NOTE 1 When the residual gas is water vapour, there is no need to characterize the interference effect.

NOTE 2 Depending on the surface area, the time to reach equilibrium could be many hours.

5.6 Residual gas analysis

- a) Total outgassing rate of the QMS in equivalent nitrogen pressure under residual pressure conditions after a bake-out and optionally outgassing rate for individual gas species of interest;
- b) sensitivity for hydrogen, methane, nitrogen and carbon dioxide at a total pressure of 10^{-5} Pa (equivalent nitrogen pressure) in a mixture of 70 % hydrogen, 5 % methane, 20 % nitrogen, 5 % carbon dioxide;
- c) fragmentation pattern for methane, nitrogen, carbon dioxide in pure gas, preferably at 10^{-5} Pa;
- d) optionally, additional sensitivities for pure gas species to be expected from the chamber may be measured, for example water vapour or dodecane as an easy-to-handle representative of hydrocarbons.

NOTE 1 Interference effect for nitrogen can be determined by comparison of sensitivity in general characterization and measurement b).

The mixture mentioned above shall prevail in the measurement chamber, see [Annex A](#).

If hydrogen is not available in the mixture described above, a separate hydrogen leak may be used to obtain the desired partial pressure, which could also be helpful for safety issues.

NOTE 2 To include water vapour in the gas mixture is desirable, but at the present stage too complicated to be realized.